

About mechanisms of radiation-induced effect of nanostructurization of near-surface volumes of metals

V A Ivchenko

Institute of Electrophysics, Ural Branch, Russian Academy of Sciences, ul.
Amundsena 106, Ekaterinburg, 620016, Russia
Yeltsin Ural Federal University, Yekaterinburg, 620002 Russia

E-mail: ivchenko2008@mail.ru

Abstract. Mechanisms of the radiation-induced development of nanostructures in subsurface metal regions have been analyzed based on field-ion microscopy data. It is concluded that the modification of near-surface metal regions on a nanometer scale as a result of the interaction with Ar^+ ion beams proceeds by several mechanisms. In particular, for a fluence of $F = 10^{16}$ ion/cm² (at an ion energy of $E = 30$ keV), the main contribution is due to the ion channeling. A tenfold increase in the ion fluence leads to prevailing deformation mechanism in nanostructure formation in the subsurface metal regions.

1. Introduction

As is known, the interaction of accelerated ions with substances leads to the formation of special condensed states in irradiated targets and, hence, unique strength and other physical properties of ion-modified materials. Experimental studies of changes in the structure of metals and alloys upon exposure [1-4] showed that ion irradiation initiates the formation of amorphous, nano-, and submicrocrystalline structures in the subsurface regions of targets. These significant changes in the structural state can lead to improvement of the physical (in particular, mechanical) properties of substances as compared to those achieved by traditional processing methods.

The results of our previous investigations [5, 6] of the surface structure of metals, carried out using the field-ion microscopy (FIM) method, showed that the irradiation of metal targets by charged particles (argon ions) with moderate energies (up to 40 keV) led to nanostructurization of a near-surface region of pure metals. Using the FIM technique, it is possible to study the subsurface layers of irradiated materials by controlled removal of atoms from the sample surface and analyze its structure in the course of layer-by-layer field evaporation of atoms. Sequential imaging of the atomically clean surface of a sample at cryogenic temperatures provides quantitative information on the positions of individual atoms and atomic groups and their changes in as a result of the implantation of positive argon ions. Thus, it is possible to study the atomic structure of radiation-induced defects in the crystalline lattice, determine their distribution in the volume, estimate the thickness of a modified subsurface layer, etc.

This work presents an attempt to analyze the effect of ion implantation on the atomic structure of platinum depending on the variable parameters (including the ion energy, ion current density, and dose) of irradiation with a beams of accelerated argon ions. The aim was to elucidate the mechanisms of formation of nanostructural states in the ion-modified subsurface region of a



target. Investigation of the laws of these phenomena is necessary for developing methods of controlled modification of the physical properties of materials, which is an important area in advanced nanotechnologies. In addition, the interaction of ions with substances provides a basis for methods of diagnostics of the initial materials and changes to them caused by ion irradiation.

2. Experimental

The targets for irradiation were made of platinum (99.99% Pt) and tungsten. The samples had the shape of point emitters with a tip curvature radius of 30-50 nm, which were made from metal wire blanks by electropolishing. Then, the samples were attested in a 1 field ion microscope to obtain images of Pt in the initial state. The attested Pt points were irradiated by a beam of 30-keV Ar^+ ions at a beam current density of $j = 150 \mu\text{A}/\text{cm}^2$ ($T = 70^\circ\text{C}$) or $j = 200 \mu\text{A}/\text{cm}^2$ ($T = 200^\circ\text{C}$) to a total fluence of $F = 10^{16}$ – 10^{17} ion/ cm^2 . The 2 ion beam was incident in the direction parallel to the axis of the point. The ion-implanted points were again placed into the field ion microscope, and sequential FIM images were recorded (by photo or video camera) during controlled removal of atomic layers. This experimental material was used for analysis of the defect structure of point samples. The field ion microscope was equipped with a micro channel ion-electron converter that enhanced the brightness of FIM patterns by 10^4 times. The cooling agent was liquid nitrogen ($T = 78 \text{ K}$), and the imaging gas was neon of spectral purity grade.

3. Results and discussion

The initial (FIM-attested) samples prior to irradiation had an atomically smooth surface of the emitter tip with a nearly hemispherical shape. This surface was obtained in situ due to field evaporation of the surface atoms. Ion images of the field emitters exhibited an almost perfect ring pattern characteristic of pure metal single crystals, which was indicative of the absence of structural defects (Figure 1a).

Figure 1b presents a typical neon image of the atomically clean surface of pure platinum irradiated with Ar^+ ions at $E = 30 \text{ keV}$ to a total fluence of $F = 10^{16}$ ion/ cm^2 . The field-ion micrographs exhibited distortions in the contrast of ring patterns of crystal face images. These distortions in the ring pattern of ion contrast revealed the appearance of defects in the perfect structure and determined a particular contrast due to defects of different types induced in the emitter by external actions. In the given case, changes in the ion contrast of irradiated platinum as compared to that of the initial (FIM-attested) metal, observed in a 1.5-nm-thick layer under the irradiated surface, are indicative of the presence of a block nano dimensional structure in the subsurface region.

Results of quantitative analysis of the distribution of block sizes in the modified subsurface region (with a volume of $V \approx 250 \text{ nm}^3$) of platinum irradiated to a fluence of 10^{16} ions/ cm^2 have been reported previously [4]. The volume fractions of nanoblocks of various sizes were determined using the Rosiwal linear method based on the Cavalieri-Acker principle.

Analysis of the ion contrast of atomic arrangement in nanoblocks (Figure 1b) showed that atoms occurred virtually in their crystal lattice sites, while blocks were misoriented relative to each other. Taking into account that the target used in experiments represented a platinum single crystal (a field ion microscope point with a tip radius of 30-50 nm is almost always single-crystalline), it can be suggested that the main mechanism of nanostructural state formation at a fluence of 10^{16} ions/ cm^2 is related to the phenomenon of channeling [7].

As a result of the irradiation to a higher fluence of $\sim 10^{17}$ ions/ cm^2 (Figure 1c), the formation of a block nanocrystalline structure (with block sizes within 1-5 nm) is observed in a near-surface layer with a thickness of no less than 20 nm under the irradiated surface.

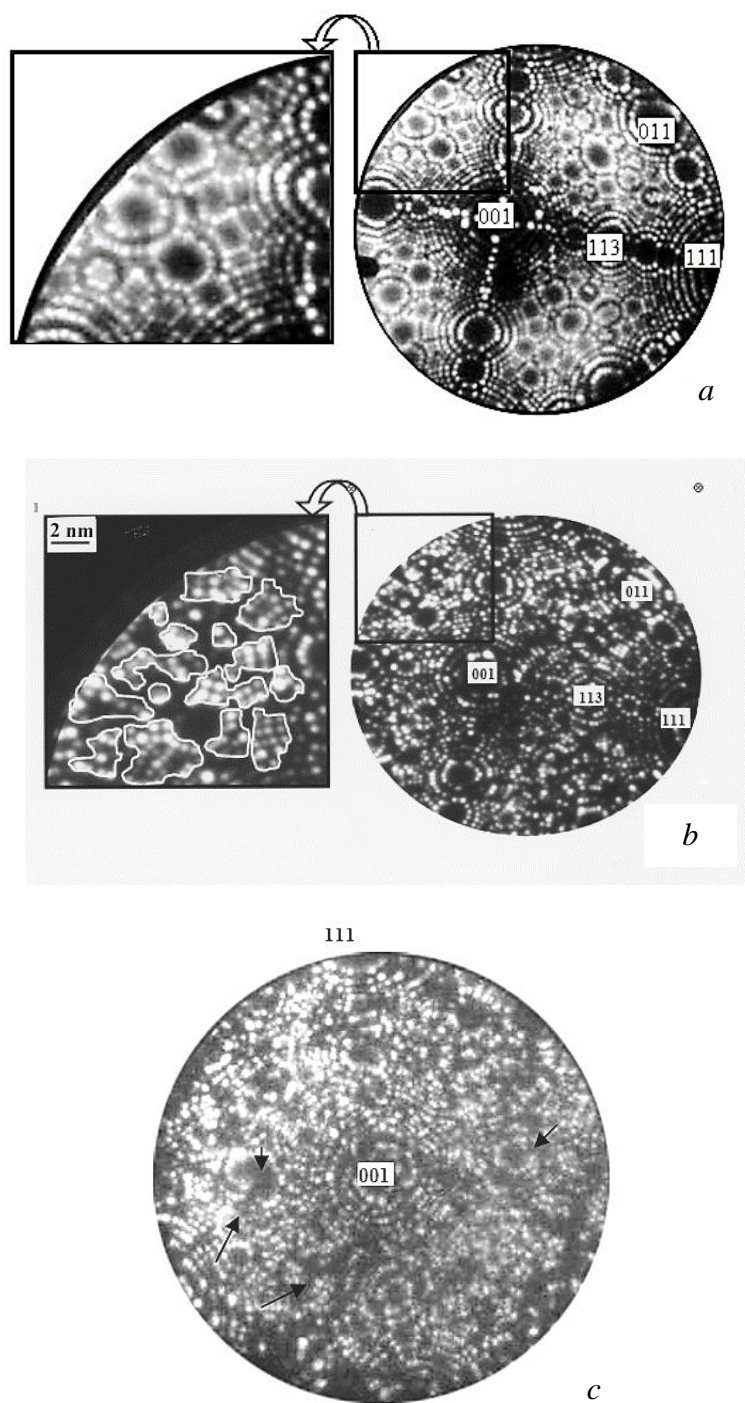


Figure 1. Neon FIM mages of Pt points (a) in the initial (attested) state, (b) upon irradiation with 30-keV Ar^+ ions to $F = 10^{16} \text{ ion/cm}^2$ ($T = 70^\circ \text{C}$), and (c) upon irradiation with 30-keV Ar^+ ions to $F = 10^{17} \text{ ion/cm}^2$ ($T = 70^\circ \text{C}$), arrows indicate the typical ion contrast of nanoparticle boundaries and stacking faults.

Analysis of the corresponding experimental data allowed the transverse and longitudinal dimensions of nanocrystalline blocks (Figure 2) and the widths of boundaries between blocks to be determined.

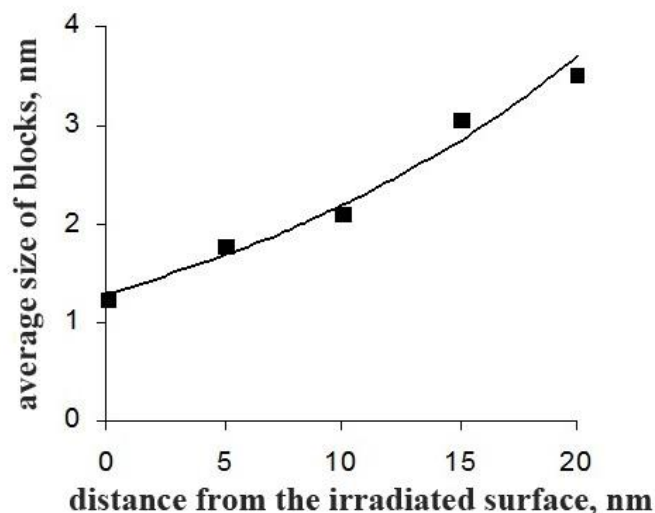


Figure 2. Variation of the average block size in depth of the transverse cross section of a sample irradiation with 30-keV Ar^+ ions to $F = 10^{17}$ ions/cm².

According to the obtained estimations, the width of these boundaries varied within 0.4-0.8 nm in various regions of nanodimensional blocks in ion-irradiated platinum points. The field-ion contrast observed on the irradiated platinum surface reveals a pattern that is typical of the grain boundaries and stacking faults [8]. This pattern is observed on FIM images of almost all faces of nanocrystals (Figure 2). This result implies that, at $F = 10^{17}$ ions/cm², the mechanism of the nanoblock structure formation in the body of the material changes.

It was shown previously [9, 10] that analogous nanocrystalline block structures could also be formed as a result of intense plastic deformation. Based on these experimental data, it can be suggested that the nanoblock structure under consideration, which appears at the stage of irradiation to $F = 10^{17}$ ions/cm² is formed as a result of deformation processes that develop in the material in the course of ion irradiation and during the subsequent period of relaxation. This hypothesis of the formation of nanostructural subsurface layer is probably confirmed by the experimental results of Didenko et al [11]. It was ascertained [11] that a surface layer modified by ion implantation is characterized by the generation of point defects, dislocation loops, and dislocations. In addition, dislocation substructures (DSSs) can form in this subsurface zone. According to the classification of DSSs [12], the ion irradiation can also lead to nanostructural states as a result of complicated evolution of dislocations.

Therefore, another possible mechanism of the formation of a nanostructural state in a subsurface layer of platinum irradiated to $F = 10^{17}$ ions/cm² is based upon the deformation model.

Thus, the effect of ion implantation on the crystal- line structure of platinum was experimentally studied on atomic spatial scale for variable parameters (including the ion energy, ion current density, and dose) of ion irradiation with a beam of accelerated argon ions. The obtained FIM data were used to analyze the possible mechanisms of nanostructural state formation in a subsurface layer within nanometer range.

It was established that modification of the crystalline lattice in a subsurface layer of ion-irradiated platinum depended on the regime of metal point irradiation with 30-keV Ar^+ ions. At a fluence of $F = 10^{16}$ ion/cm², the observed nanostructurization of the subsurface layer is most probably related to the phenomenon of channeling.

A tenfold increase in the ion fluence led to a change in the mechanism of nanostructural state formation in the irradiated region. The deformation character of the ion contrast observed on the metal surface during controlled sequential removal of atomic layers, followed by an analysis of the state of the near-surface layer using the experimental FIM data, led to the conclusion that the deformation mechanism plays a dominant role in the formation of nanostructure in the subsurface layer. It can also be expected that the formation of a nanostructural state in a near-surface layer of ion-irradiated materials can lead to a significant increase in their physical (in particular, mechanical) properties.

Acknowledgments

This work was performed as part of State Task No. 0389-2014-0002, was supported in part by RFBR Grant No. 15-08-06744.

References

- [1] Guseva M I and Gordeeva G V 1986 *Phys. Status Solidi A* **95** 385
- [2] Ovchinnikov V V 1996 *Izv. Ross. Akad. Nauk: Metall* **6** 104
- [3] *Surface Modification and Alloying by Laser, Ion and Electron Beams* 1983 (Ed. by Poate J M, Foti G and Jacobson D C; New York: Plenum)
- [4] Ivchenko V A and Medvedeva E V 2009 *Izv. Vysch. Uchebn. Zaved. Fiz.* **8/2** 410
- [5] Ivchenko V A and Medvedeva E V 2009 *Perspekt. Mater* **7** 119
- [6] Ivchenko V A and Medvedeva E V 2010 *Bull. Russ. Acad. Sci.: Phys.* **74** 2 217
- [7] Nastasi M, Mayer J W and Hirvonen J K 1996 *Ion-Solid Interactions: Fundamentals and Applications* Cambridge Solid State Science Series, **27** (Cambridge: Cambridge University Press)
- [8] Boowkett K M and Smith D A 1970 *Field Ion Microscopy Defects in Crystalline Solids* (Amsterdam, London: North Holland Publishing Company)
- [9] Ivchenko V A and Syutkin N N 1999 *Pis'ma Zh. Tekh. Fiz.* **25** 6 410
- [10] Ivchenko V A, Efros B M, Popova E V, Efros N B and Loladze L V 2003 *Fiz. Tekh. Vys. Davlen.* **13** 3 109
- [11] Didenko A N, Sharkeev Yu R, Kozlov E V and Ryabchikov I 2004 *Long-Range Interaction Effects in Ion-Implanted Metal Materials* (Tomsk: Izdat. NTL)
- [12] Koneva N A, Kozlov E V and Trishkina L I 1991 *Metallofizika* **12** 149